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Diamond(100) was studied as a substrate for the adsorption of atomic hydrogen and fluorine. Both surface C-H bonds and surface C-F bonds are of importance in the tribological use of diamond film coating. A new and facile method for depositing surface fluorine on diamond has been developed. The method involves the photochemical dissociation of perfluoroalkyl iodide molecules depositing C_nF_{2n+1} groups on the diamond. These groups, upon thermal dissociation, produce surface C-F groups on the diamond. A patent application is being researched on this new method.

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PRINCIPAL INVESTIGATOR: John T. Yates, Jr.

INCLUSIVE DATES: March 15, 1992 - March 14, 1995

CONTRACT/GRANT NUMBER: F49620-92-J-0192

SENIOR RESEARCH PERSONNEL: Dr. Jian Wei (to April 1994)
Dr. Vincent S. Smentkowski
(August 1994 - March 1995)

JUNIOR RESEARCH PERSONNEL: Vincent S. Smentkowski
Thomas Barefoot

PUBLICATIONS:

Twenty papers have been published or submitted as a result of this contract. They are listed below:

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465. V.S. Smentkowski, J.T. Yates, Jr. and W.H. Goddard III, "Fluorination of Diamond - C_4F_9I and CF_3I Photochemistry on Diamond(100)", in preparation.

ABSTRACT OF OBJECTIVES AND ACCOMPLISHMENTS:

Diamond(100) was studied as a substrate for the adsorption of atomic hydrogen and fluorine. Both surface C-H bonds and surface C-F bonds are of importance in the tribological use of diamond film coating. A new and facile method for depositing surface fluorine on diamond has been developed. The method involves the photochemical dissociation of perfluoroalkyl iodide molecules depositing C_nF_{2n+1} groups on the diamond. These groups, upon thermal dissociation, produce surface C-F groups on the diamond. A patent application is being researched on this new method.

Surface Chemistry of Diamond Single Crystals
1992-1995

I. Objectives and Summary of Progress

The goals of this project are to measure the surface behavior of diamond (100) as it interacts with functionalizing species to produce surface C-H and C-F bonds. Work on C-H bonding has been completed and a paper on this subject has been accepted for publication (paper 450, see end). We find that the C_{1s} spectrum is indicative of surface and bulk disordering as atomic H reacts with the surface breaking C-C bonds and forming C-H bonds. Experimental work on C-F bond formation is also completed, using CF_3I and C_4F_9I as reactive sources of C-F bonds at the surface. Such studies provide insight into surface chemical processes at work in diamond CVD film growth as well as in tribological areas involving diamond sliding interfaces. A definitive review of "Surface Chemistry of Diamond" has been accepted by Critical Reviews in Surface Chemistry. Along with this narrative review, an extensive bibliography, containing over 900 references on diamond surface chemistry will comprise an accompanying volume of Critical Reviews in Surface Chemistry.

In addition, work defining methods for the accurate measurement and control of diamond crystal temperature has been published, and definitive studies of conventional atomic O sources involving heated Ir and Pt filaments have been published. It was shown that almost all published work on diamond tends to over estimate the diamond temperature (sometimes by several hundred K). It was also shown that commonly employed Ir and Pt hot filament sources for atomic O do not deliver pure atomic O to surfaces, but instead deliver metal impurities mixed with oxygen.

II. Progress - Selected Examples

A. Diamond Crystal Heating

We, as well as others, have had difficulties with heating diamond crystals by radiation to temperatures above 1400 K; thus, an enhanced heating and temperature measurement procedure has been designed in which a combination of radiation and electron bombardment is employed. Care has been taken to avoid

any exposure of the crystal to electrons by using a shielded source in the indirect heater. This method has been shown to be successful, and a paper has been published on this method (V.S. Smentkowski and J.T. Yates, Jr., J. Vac. Sci. Technol. A11 (1993) 3002).

Figure 1 shows that we have been successful in heating the diamond reproducibly and that the use of embedded thermocouples to measure the true temperature of the diamond is far superior to measurements of the support temperature alone. Errors of hundreds of degrees K may be made by measuring the support temperature. This method of directly measuring the diamond temperature is unique to this laboratory as all other workers to date have employed only the support-temperature measurement method.

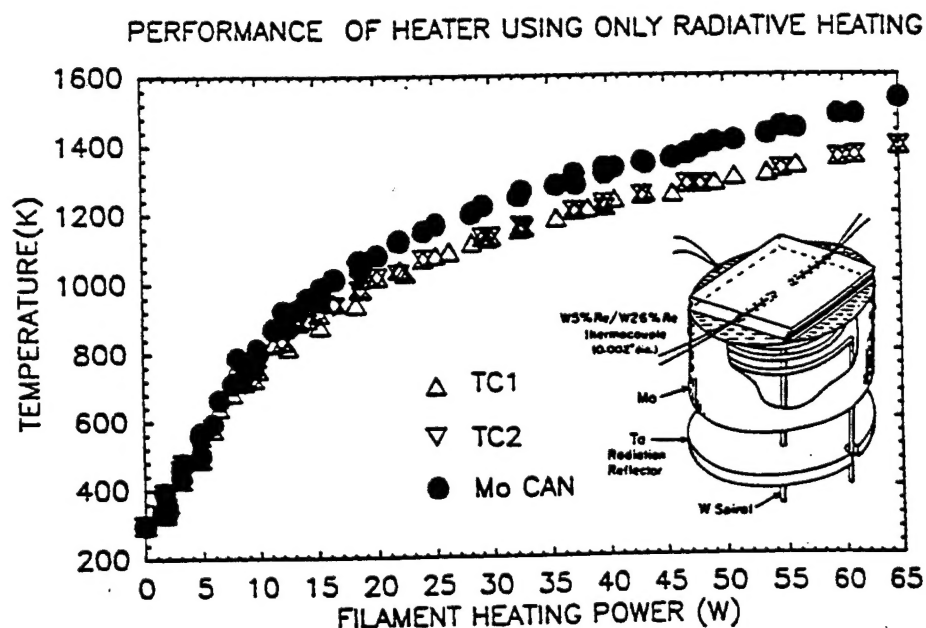PERFORMANCE OF HEATER USING ONLY RADIATIVE HEATING

Figure 1. Comparison of true diamond temperature and support temperature during steady-state heating of diamond using the mounting arrangement shown in the inset.

Figure 2 illustrates a programmed heating experiment for the diamond crystal. It may be seen that: (1) the two diamond thermocouples agree to within 15 K, and (2) at a diamond temperature of 1400 K, the Mo support can is ~225 K hotter than the diamond.
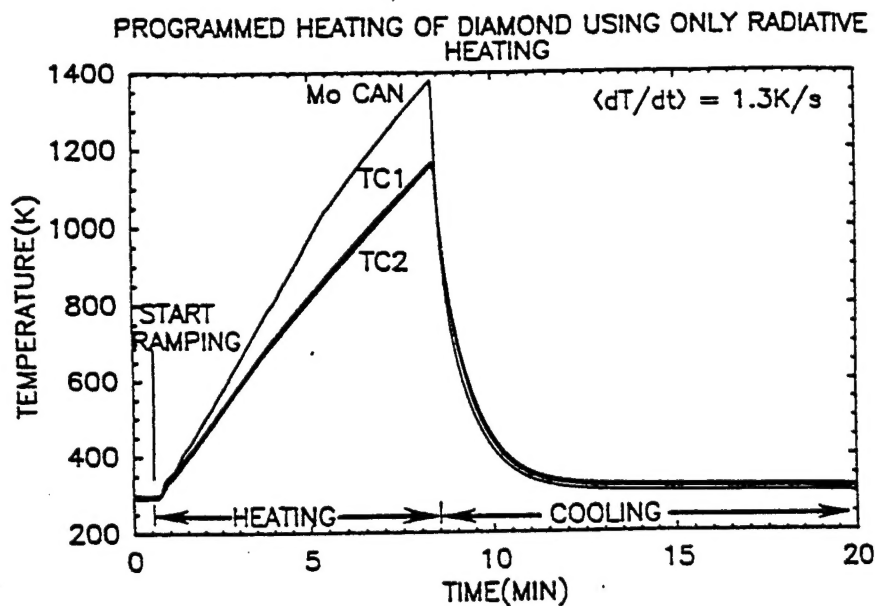
PROGRAMMED HEATING OF DIAMOND USING ONLY RADIATIVE HEATING

Figure 2. Programmed heating experiment. The temperature programmer is adjusting its output based on the temperature measured inside the diamond.

B. Atomic Oxygen Sources - Ir and Pt Filaments

The use of atomic oxygen to oxidize the diamond was an objective of this work, since atomic oxygen may lead to novel surface oxidation products. There are reports in the literature of the use of both hot iridium as well as hot platinum filament surfaces operating in O_2 gas as a source of atomic oxygen. Prototype sources of this type have been constructed and thoroughly studied by us using two methods (gas phase analysis and surface trapping analysis) to detect atomic O. It has been shown that no atomic O is produced by this source without the

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production of unacceptable fluxes of metallic vapor (Ir and Pt) which is transported to the surface.

Figure 3 shows conclusively that atomic O does not transfer to a Au test surface without the transport of iridium also, as determined by Auger spectroscopy measurements made in this laboratory. This work therefore demonstrates that previous published studies made elsewhere of the oxidation of diamond by atomic oxygen produced on a hot iridium filament are false. A paper on this work has been published (V.S. Smentkowski and J.T. Yates, Jr. J. Vac. Sci. Technol. A12 (1994) 219).

GOLD TRAPPING EXPERIMENTS BY AUGER SPECTROSCOPY AS A FUNCTION OF Ir TEMPERATURE

OXYGEN EXPOSURE = 2,000 L

Figure 3. Study of the transport of iridium and atomic oxygen to a Au test surface from a hot Ir source operating in O_2. Note that before detectable atomic O is collected, Ir is transported at an Ir temperature of about 1500-1600 K.

Figure 4 shows similar experiments using a heated Pt filament. Again, it is demonstrated that atomic O does not transfer to a gold test surface without the transport of Pt - indicating that heated Pt filaments can not be used to generate pure atomic oxygen - as discussed fully elsewhere (V.S. Smentkowski and J.T. Yates, Jr., J. Vac. Sci. Technol. A12 (1994) 224).

7

GOLD TRAPPING EXPERIMENTS BY AUGER SPECTROSCOPY
AS THE Pt FILAMENT TEMPERATURE IS CHANGED

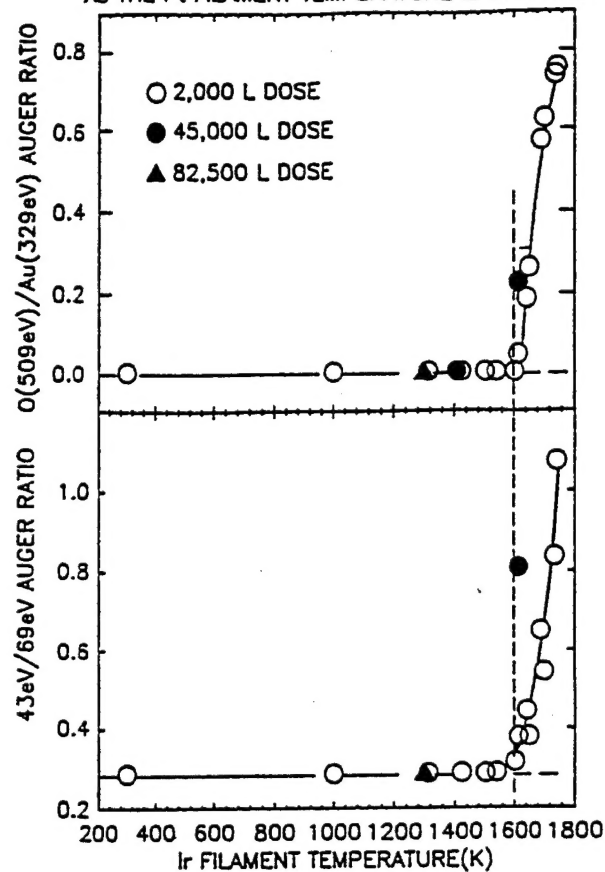O 2,000 L DOSE
● 45,000 L DOSE
▲ 82,500 L DOSE

Figure 4. Study of the transport of platinum and oxygen to a gold test surface from a hot Pt filament operating in O_2.

As neither of these methods could be utilized to produce pure atomic oxygen, we have discontinued our plan to treat the diamond with atomic O.

C. Graphite Detection and Removal from Diamond

Diamond is the unstable form of carbon (the free energy of formation of diamond is 0.693 kcal/mole greater than graphite). The XP spectra of graphite

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exhibits a $\pi \rightarrow \pi^*$ transition resulting from a shake-up transition, at approximately + 6 eV above the C_{1s} binding energy. This shake-up transition is not observed for freshly polished diamond as shown in the upper spectrum of Figure 5. The lower spectrum of Figure 5 shows the diamond surface with graphite present as indicated by the graphite shake-up transition. To the left of this plot, is an energy schematic which illustrates the involvement of the shake-up transition.
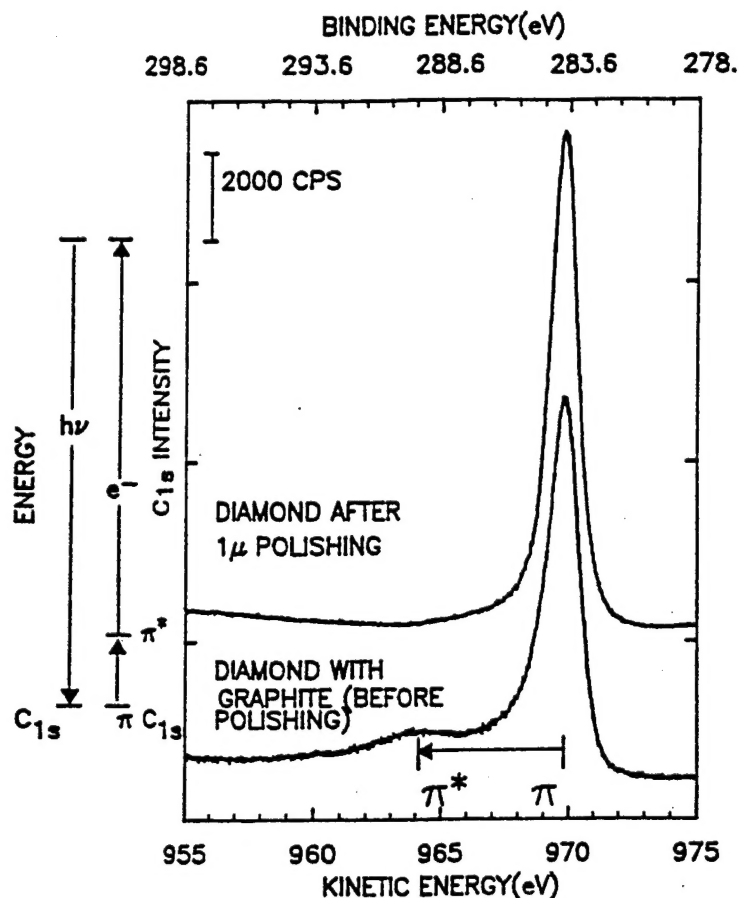

Figure 5. **XP spectra of diamond(100); top spectrum corresponds to a freshly polished surface while the bottom spectrum corresponds to a diamond surface with surface graphite present.**

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These data therefore indicate that XPS is capable of distinguishing between graphite and diamond. It is not known whether the graphitization resulted from accidental electron beam exposure or accidental high temperature heating during our initial experiments.

We tried to chemically etch the graphite away using both, atomic D (72,000 L at 700 K) and molecular O_2 (2,000 L at 1250 K) under UHV conditions. Neither of these methods were successful and the diamond was therefore repolished.

D. Initial Heating of Freshly Polished Diamond (100)

Figure 6 shows representative C_{1s} spectra as the diamond is initially heated to 1450 K. Dramatic spectral shifts and broadening effects are observed on first heating the diamond.
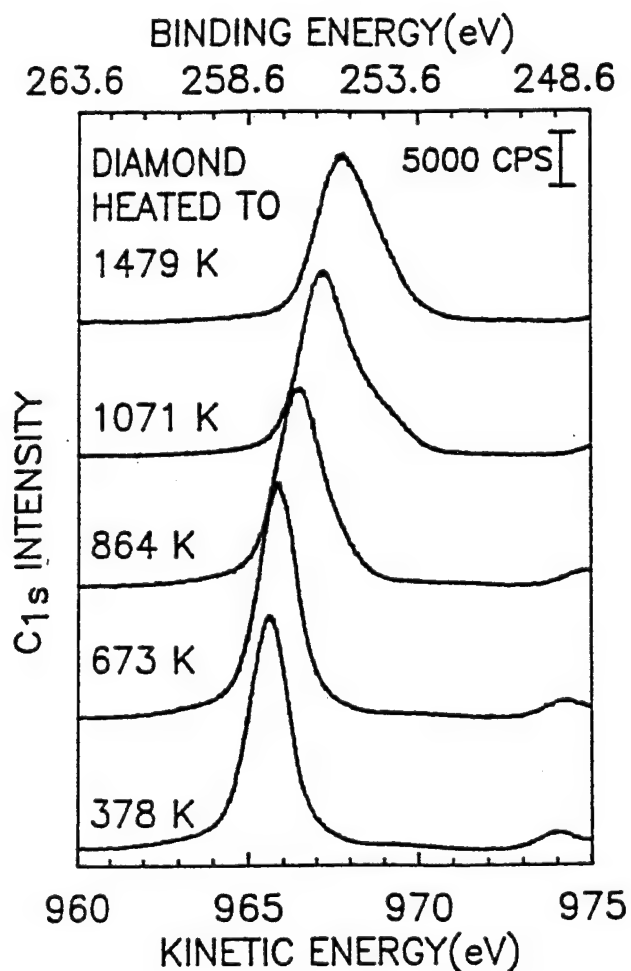

Figure 6. C_{1s} spectra of diamond obtained upon the initial heating.

As the diamond is sequentially heated (in steps of ~ 50 K) to about 700 K, the peak width and binding energy of the C_{1s} XPS transition remains almost unchanged. Further heating to 850 K results in: (1) broadening of the C_{1s} transition as well as, (2) a shift of the peak maximum to lower binding energies. At a temperature of ~ 1135 K, the LEED pattern changes from a (1x1) to a (2x1)/(1x2) structure - indicating that the C(100) surface is starting to reconstruct. As the temperature is further increased to 1450 K, the C_{1s} transition shifts to still lower binding energy while the (2x1)/(1x2) LEED pattern intensifies. Although the C_{1s} transition is broadening, the total integrated area of the C_{1s} feature remains essentially constant. An O_{1s} XPS signal decreases in the temperature range studied here, indicating that the surface of the diamond is oxidized following preparation before this experiment, and that high temperature desorption of the oxide layer occurs.

These observations may be explained as being due to: (1) improved contact between the diamond (100) crystal and the Mo support can, as well as (2) removal of surface oxidation products as the diamond is initially heated.

E. Atomic Deuterium Interaction with Diamond (100)

The interaction of atomic deuterium with diamond (100) has been studied by X-ray photoelectron spectroscopy (XPS). As the diamond surface is exposed to atomic deuterium, the FWHM of the C_{1s} transition initially broadens for low atomic deuterium exposures as shown in Figures 7 and 8. Increasing the atomic deuterium exposure to higher values results in a sharpening of the C_{1s} transition. By performing the complementary experiment of dosing the diamond to high exposures and heating stepwise, it is demonstrated that this process is essentially reversible. This broadening phenomenon is postulated to be due to the production of partially deuterated carbon atoms in the surface and near surface regions as C-D bonds form from D atom insertion into the C-C back bonds. Continued D atom exposure is postulated to produce a more uniform distribution of C-D bonds as more complete deuterium incorporation takes place - as schematically illustrated in Figure 9.
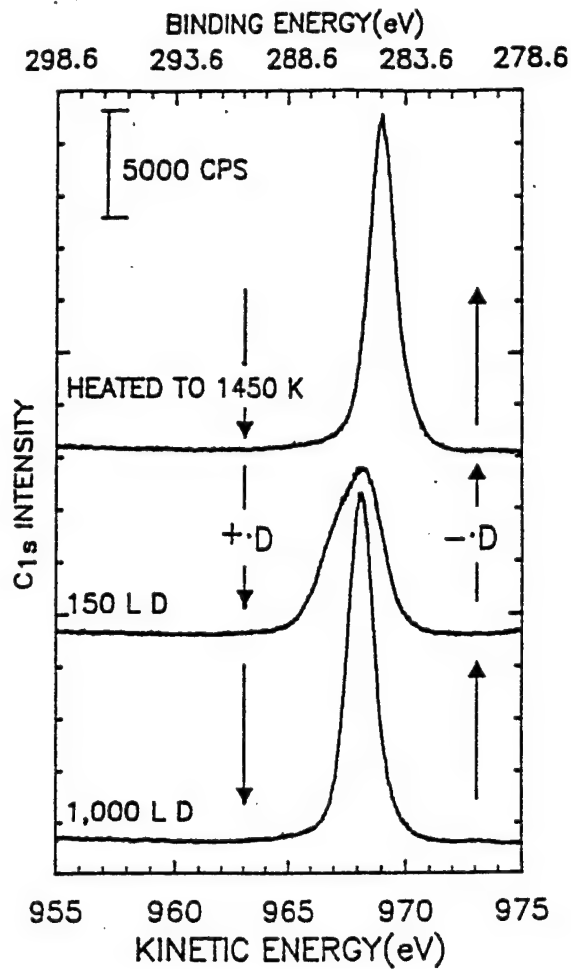
Figure 7. C_{1s} spectra of diamond following atomic deuterium exposure.
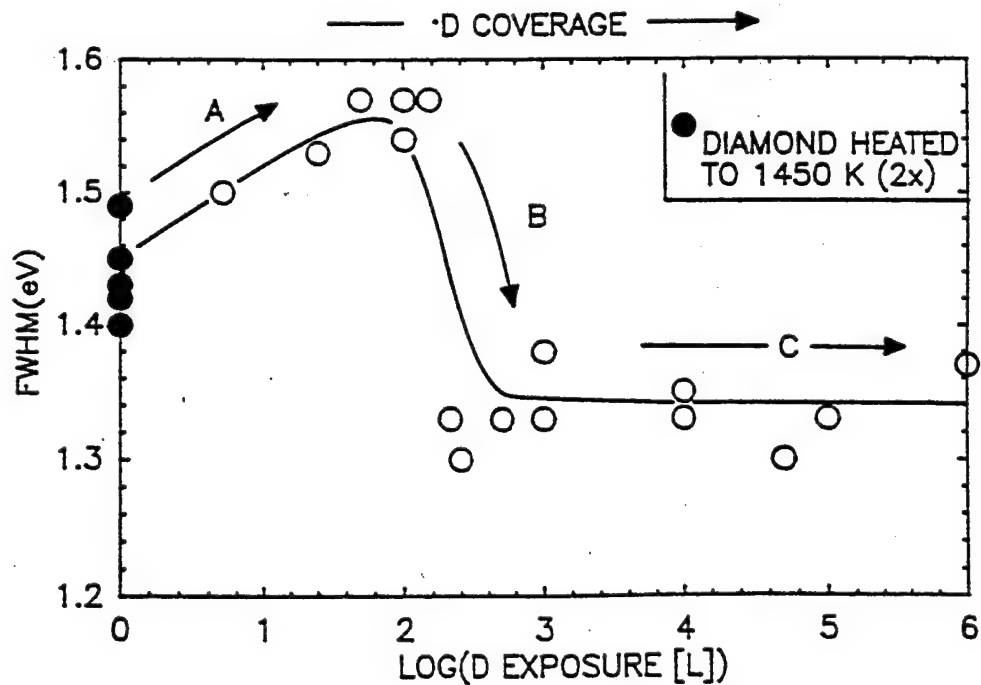

Figure 8. Plot illustrating the change in FWHM of the C_{1s} XP feature as a function of atomic deuterium exposure.
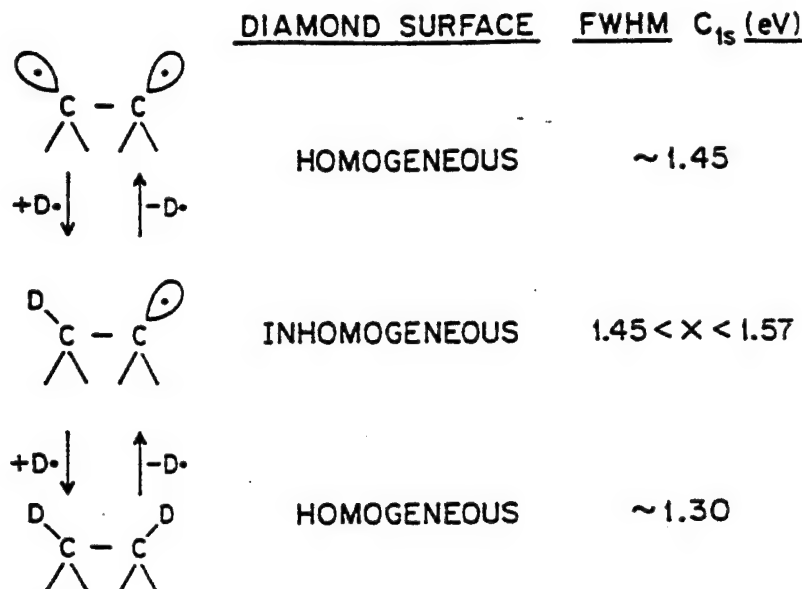
DIAMOND SURFACE   FWHM C$_{1s}$ (eV)

HOMOGENEOUS   ~1.45

INHOMOGENEOUS   1.45 < X < 1.57

HOMOGENEOUS   ~1.30

Figure 9. Schematic illustrating a potential mechanism for the change in FWHM of the C$_{1s}$ feature during the interaction of atomic deuterium with diamond(100).

F. Easy Fluorination Route for Diamond

We have discovered a very easy fluorination route for diamond(100) using the photochemical dissociation of the C-I bond in fluoroalkyl iodides. Both CF$_3$I and C$_4$F$_9$I have been used for this process, and it has been shown that large fluorine coverages may be achieved by use of the higher molecular weight fluoroiodide molecules. Figure 10 shows the XPS spectra of the diamond(100) surface covered with a weakly-held C$_4$F$_9$I layer at 119 K. Irradiation, followed by heating to 300 K to desorb the unreacted C$_4$F$_9$I layer leaves evidence for surface F in the form of CF$_2$ groups and CF$_3$ groups, as seen in the F(1s) and C(1s) spectral regions.
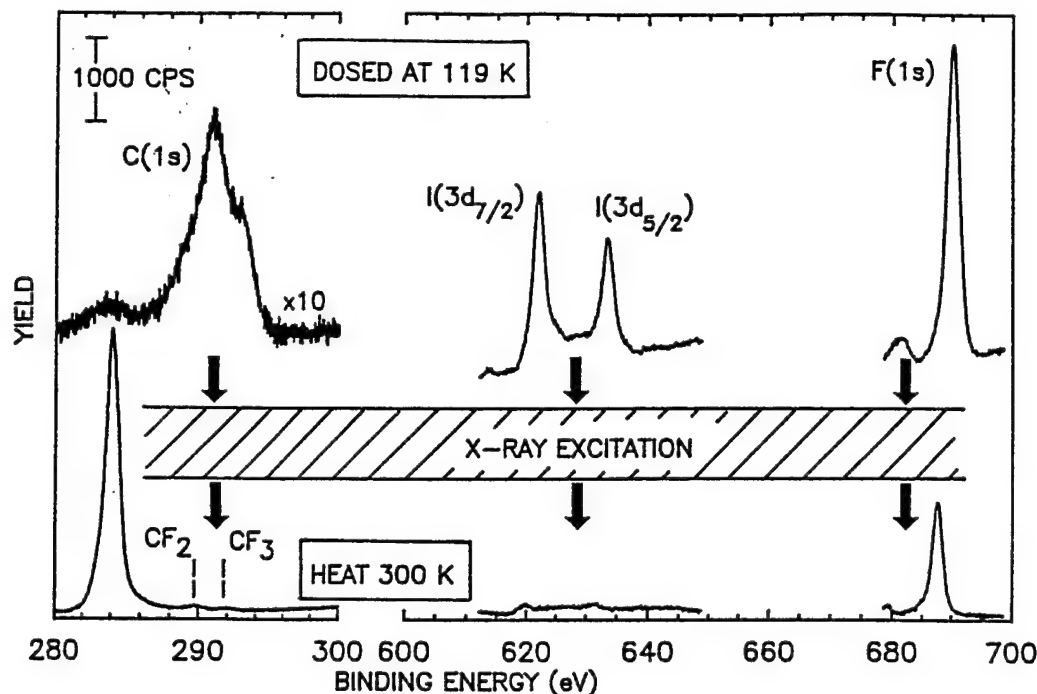
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Figure 10. C$_4$F$_9$I Condensation and Photochemically-Induced Chemisorption on Diamond(100).

The production and loss of surface F and surface I following the photochemical anchoring of C$_4$F$_9$ groups to the diamond surface is shown in Figure 11. The surface F resulting from C$_4$F$_9$ thermal decomposition is present on the surface up to 1500 K.
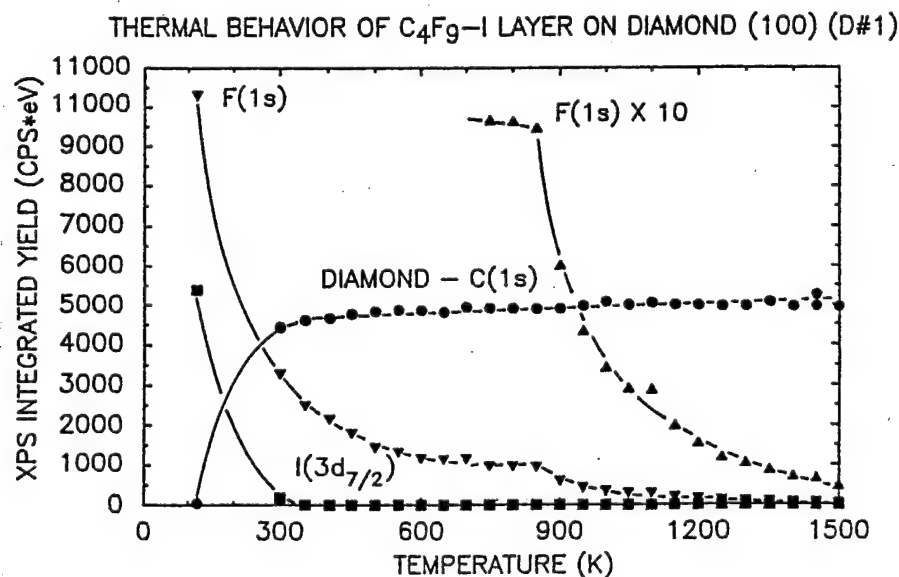

Figure 11. Thermal Behavior of C$_4$F$_9$-I Derived Layer on Diamond(100).

A high sensitivity measurement of the C(1s) behavior during heating in vacuum shows that both CF_2 and CF_3 groups are observed to decompose over the temperature range 300 K-700 K. This is indicative of the presence of C_4F_9 groups on the surface in this temperature range.

The behavior of the C_4F_9 groups is seen in Figure 12.

CHANGES IN CF_2 AND CF_3 XPS FEATURES
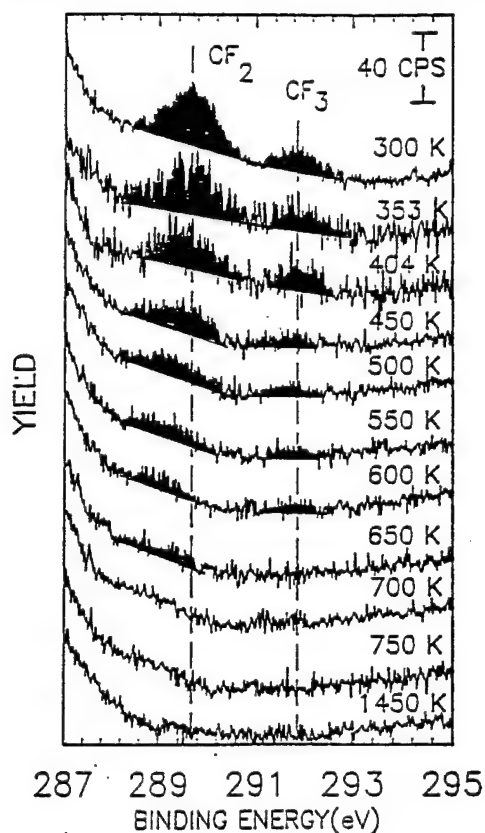UPON HEATING C_4F_9I/DIAMOND (100) (D#1)

Figure 12. Changes in CF_2 and CF_3 XPS Features upon Heating.

When thermal decomposition occurs, F is captured by the diamond surface, and the surface F groups are found to be stable up to about 1500 K, as shown in Figure 13. Also, shifts in the F(1s) binding energy may be used to distinguish between fluorine in CF_2 or CF_3 groups from chemisorbed F on the diamond surface itself, as indicated in the figure.
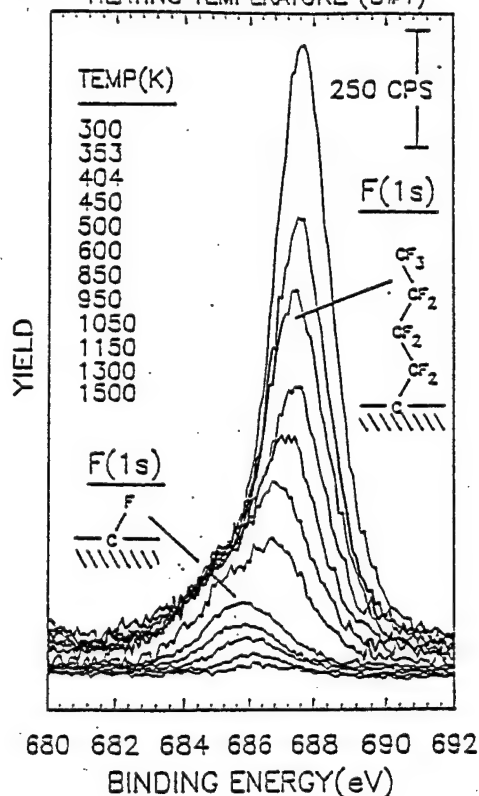
Figure 13. Changes in F(1s) XPS Intensity as a Function of Heating in Vacuum.

We found that the chemisorbed F produced by this photochemical procedure, followed by heating to decompose the fluroalkyl group, exhibits a high level of thermal stability. Figure 13 illustrates this property, where it is observed that the surface fluorine begins to desorb at about 500 K, but is still detectable to near 1500 K. In Figure 13, the dark points are obtained by ourselves, whereas the white points were obtained by A. Freedman at Aerodyne, using an atomic F source to deposit F directly on diamond(100).

The addition of fluorine to diamond(100) was found to occur by the filling of the half-filled surface states. Since this results in the elimination of surface charge, the bands in the diamond are flattened. This causes the C(1s) level to increase in binding energy as a result of the band flattening due to F chemisorption. Figure 14 illustrates this shift of the C(1s) level for surface atoms as well as for atoms in the near surface region which are pinned to the band edges. Such shifts in the C(1s) binding energy amount to about 0.5eV for our natural diamond samples, and may be used to monitor the degree of surface fluorination.
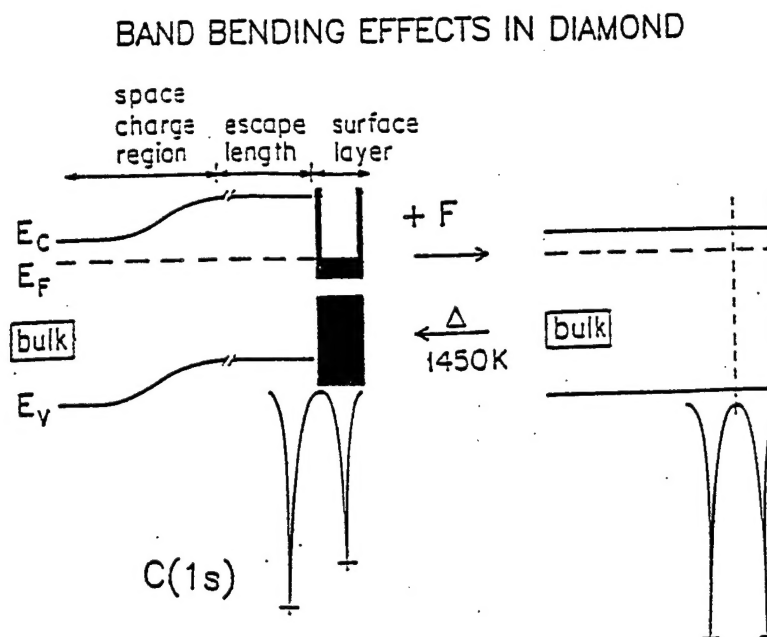
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BAND BENDING EFFECTS IN DIAMOND

Figure 14. Band Bending Effects in Diamond.

These results therefore illustrate an easy route to the fluorination of diamond surfaces. X-ray irradiation, which was used here, is probably not necessary for activating this chemistry, since the C-I bond is photochemically broken with ultraviolet light.

The method outlined above is the subject of a patent application being prepared by the University of Pittsburgh.

III. Personnel

Graduate Students - Mr. Vincent S. Smentkowski
Ph.D., August 1994

Mr. Thomas Barefoot

Postdoctoral - Dr. Jian Wei (to April 1994)
Dr. Vincent S. Smentkowski
(August 1994 - March 1995)

IV. Papers Published or Submitted to March 15, 1995

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